NASA CR-72459

PREPARATION OF FILAMENT WOUND GLASS MICROTAPE RESEARCH SPECIMENS

PREPARED FOR

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER

D&R

DE BELL & RICHARDSON, INC

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Contract No. NAS3-3647

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HAZARDVILLE, CONN. 06036

July 5, 1968

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SUMMARY

The objective of this project was the forming of low permeability, thin-walled tubes suitable for containing cryogenic liquids, with no liner, from rectangular glass fibers and resin.

Through the application of the preform attenuation process for making shaped glass fibers, high glass content, cylindrical structures were filament wound from a single wide, thin, flat, uniform microtape of glass.

In order to achieve these results, it was necessary to refine the filament forming process to produce glass filaments with a high aspect ratio, flat rectangular cross section. A guiding and precision winding technique had to be developed to obtain the desired edge-to-edge, flat layered structure. An acid polishing process was worked out to provide low surface flaw count preforms from which to draw higher strength microtape. A means of dynamically monitoring the microtape width was developed to assist in precision winding.

The test results on finished cylinders showed that the structure had a modulus of elasticity virtually equal to that for the glass. Strengths measured by internal burst and straight axial tensile, gave results below the modest strength of the microtape filament. It is indicated that a resin and coupling agent system which would bond better and have higher elongation should be developed to fully utilize the potentialities of glass microtape filament wound structures while the strength of the microtape itself should be further improved.

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PREPARATION OF FILAMENT WOUND MICROTAPE RESEARCH SPECIMENS

INTRODUCTION

Lewis Research Center had shown that glass-fiber-reinforced plastics have good mechanical properties at cryogenic temperatures. This feature makes these composite materials attractive for fabricating cylindral, cryogenic, liquid-propellant containers. Unfortunately, the minute hydrogen molecule can readily permeate through the resin phase with its microcracks, indicating a liner would be necessary.

The problem, therefore, was to develop a wide, thin, flat microtape which should minimize permeability through a properly wound structure. This would circumvent the need for a liner and could possibly, at the same time, provide extra properties not available with round filaments or strands presently used in filament winding.

Glass microtape is a continuous, solid tape of microscopic dimensions. Figure 1 attempts to illustrate this definition. Most commercial glass fiber is about 0.00037 in. in diameter. Microtape is a continuous, minute glass tape whose thickness may approximate the diameter of common glass filament but whose width can be made to be 35 to 50 or more times its thickness.

THEORETICAL CONSIDERATIONS

An examination of leakage paths through composite structures is important to visualize the potential advantage of microtape wound structures for low permeability container walls. Permeability through a glass fiber reinforced structure is inversely proportional to the leakage path length, and approximately proportional to its cross-sectional area and the number of leakage paths per unit area. Permeability of glass to gases is neglibible, especially at low temperatures; only the resin phase needs to be examined.

Round fibers, even in perfect hexagonal packing as shown in Figure 2, present a leakage path length L of $\pi/3$ times (only 1.05 times) the wall thickness t of the vessel under consideration.

It is easy to see how microtape can extend the leakage path length. Consider a microtape whose width-to-thickness ratio R is 50 and is wound in flat layers in close juxtaposition to one another. If this is staggered in winding so that a microtape is centered exactly over the joint in the underlying layer, as shown in the model (Figure 3), the leakage path becomes

$$L = \left(\frac{R}{2} + 1\right) t$$

$$L = \left(\frac{50}{2} + 1\right) t = 26t$$

This assumes the number of layers of microtape is large, as is the case with the thin microtape under consideration. Thus, in the same wall thickness, the 50:1 microtape presents a leakage path which is 25 times as long as that for perfectly packed round fibers. Even a 1/3 overlap (Figure 4) provides a path length over 19 times that for round fiber. A purely random winding of microtape side by side will provide a leakage path which is equivalent to a 1/4 overlap. The path length then becomes

$$L = \left(\frac{R}{4} + 1\right) t$$

$$L = \left(\frac{50}{4} + 1\right) t = 13.5t$$

This is still over 12 times the path length of a perfectly packed round filament composite.

Another difference found between a round fiber composite and one made from microtape is the cross-sectional area of the leakage path. Getting back to the well-packed round filament composite (Figure 2), the cross section of the leakage path is related to the size of the filaments. For discussion purposes, consider a standard commercial fiber of 0.00037 in. The area the gas molecule "sees" is at right angles to the one seen when studying a section through the wall and the ends of the filaments. One of its dimensions is the circumference of the cylindrical vessel and is the same for any shaped filament. The other dimension will average no less than 1/8 the filament diameter or 0.000046 in. To arrive at this estimate, the tricorns where three round filaments are nearly in contact were calculated as equilateral triangles, and the average altitutde of such a triangle was estimated to be 1/8 the diameter of the filaments.

With a good quality filament winding of microtape, on the other hand, a resin layer between flats of microtapes of less than 1/4 μ, or less than 0.00001 in., has been measured.

A more realistic appraisal of existing conditions in winding of round filaments would be to assume an approach to square packing (Figure 5) which gives a path length equal to the wall thickness and an average resin layer

through the quadricorns approaching 1/3 the diameter of the filaments, or 0.00012 in.

Thus, a comparison, on paper, of permeability of 50:1 microtape composite with optimum filament winding practice shows the path length to be at least 12 times longer for microtape and a resin layer over 12 times thicker in round filament windings. The number of leakage paths through a unit area of wall is at least 50 times greater in a round filament-wound structure than it is with 50:1 microtape. Therefore the permeability of a microtape structure is predicted to be less than one made from round filament by the following factors because of the different path characteristics:

Length		Thickness		Frequency		
$\frac{1}{12}$	X	$\frac{1}{12}$	X	$\frac{1}{50}$	=	$\frac{1}{52,200}$

In other words, the permeability of a microtape structure should be only 1.9×10^{-5} times that for an ordinary filament-wound vessel, all other things being equal.

OBJECTIVES

With these theoretical advantages in mind, it was planned that one filament wind, a Pyrex borosilicate, or other suitable composition glass microtape 3 to 10 microns thick with a width-to-thickness ratio of as much as 50:1 or even 100:1. An analysis of microtape single filament strength was also spelled out as part of Phase I of the program. This microtape was to be biaxially wound into 5-1/2" diameter 16" long test cylinders. It was also the plan to apply coupling agent to the filament from the vapor state and then apply a solvent dispersed epoxy resin which would be B-staged prior to winding on a forming tube. This microtape was then to be wound into the 5-1/2" cylinders in close edge-to-edge fashion to produce the desired high glass content, low permeability wall structure.

The plan also called for NOL type hoops and flat tensile specimens to be made before the tubes. The former were to test the strength of the microtape as wound while the latter were to test the tensile strength of the structure normal to and in the plane of the microtape. The design of the flat tensile specimens was changed to micro-tensile specimens to permit cutting them axially from a cylindrical band so the structure would be identical to the as-wound material.

As the program proceeded, a number of changes were indicated and these were subsequently made by mutual agreement.

The borosilicate composition had been suggested since it is more like E-glass, commonly used to make glass fibers, than is common soda-lime glass and Pyrex is available in sheet form. However, because of the tendency of Pyrex borosilicate glass to devitrify and thereby lose strength at the forming temperatures used in DeBell & Richardson's preform attenuation process, soda-lime window glass was used throughout the program. Microtape drawn from Pyrex did display reduced tensile strength.

Many of the earlier specimens in the program were made from filaments about 10 microns thick. As the winding experiments proceeded, the filament placement problem made it obvious that larger rectangular cross section filaments were necessary to guide them accurately into place. The final tube specimens submitted to Lewis Research Center were made from filaments almost 40 microns thick and proportionately wider.

Although it was demonstrated that microtape could be made with a width-to-thickness ratio of between 50:1 and 100:1, it was found that crisp, rectangular cross sections could best be formed if the ratio was held to about 40:1. The microtape which was used in the later specimens with good placement were about 0.060 x 0.0015". When small diameter (2.300" I.D.) cylinders were pressure tested it was found that respectable strength valves were achieved even though no axial or geodesic windings were applied. The overlap of even 40:1 microtape gave surprising axial strength, so biaxially wound structures were postponed to have been taken up later in the program.

Thus, in order to produce the filament wound glass microtape research specimens, three separate problems had to be solved, solved individually at first and then accomplished simultaneously. First, high aspect ratio microtape had to be drawn that was virtually flat with no edge beads and with precise thickness and width dimensions. Secondly, the tape had to be placed in an accurate helix in close edge-to-edge fashion to form essentially a single cylindrical surface for each layer of microtape wound. Finally, the microtape had to be bonded with a resin having a group of requirements somewhat unusual for resin esystems for filament winding.

DISCUSSION

Process Description

The preform attenuation process developed at DeBell & Richardson, Inc. and used to form microtape has been described in some detail in various places. NASA CR-142(1) contains such a description. A new book (2) contains a chapter entitled "Glass Microtape" which more specifically describes the details of forming microtape. A paper (3) was presented before the

VIIth International Glass Congress in Brussels on July 1st of 1965 on shaped fiber forming.

Very briefly, a preform is fed slowly downward into a small furnace whose temperature permits the glass to soften. The softened glass is then stretched or attenuated into a filament and carried out of the bottom of the furnace, without pulling through a die, to form a glass fiber whose cross sectional shape is similar to the piece of glass or preform being fed slowly down into the furnace. The reduction in size of the preform producing the filament might be as much as 100 times in each dimension which would necessitate a drawing rate of approximately ten thousand times the feed rate.

The problem, then, becomes one of maintaining or producing the desired cross sectional shape in the filament; in this case, a thin, glass, continuous filament called microtape, approximately 0.0005" thick and 0.020" or more wide. The preform used to make microtape is a thin sheet of window glass (photo glass) about 0.050" thick, 3" wide and about 4' long. Secondly the microtape must be drawn to very constant dimensions in order to make a perfect lay up in an edge-to-edge fashion. The forming then of such a precisely shaped and dimensioned filament is one vital phase of this project.

Once a suitable filament is formed, it must be placed in exactly the right place on the mandrel in order to filament wind a structure which may have the desired low permeability. This requires the winding of a perfect, regular helix on a cylindral mandrel in such a way that the filament is placed edge-to-edge throughout each layer. Thus, the mandrel or the filament guide must move linearly in an axial direction at exactly the proper rate to advance one filament width, plus any permissible gap, with each revolution of the mandrel. Therefore, another phase of the project which must be accomplished in order to wind suitable research specimens is the precision winding of microtape.

The chapter on "Glass Microtape" (2) describes the evolution from the first approach which had produced good equidimensioned shaped fibers to the furnace designs which produced more nearly perfectly formed microtape. That is, the early work on shaped fibers had been most successful with a furnace having essentially a pancake-shaped hot zone through which the preform was lowered. On the other hand, a study of the dynamics of microtape drawing indicated that the wide flat preform required a gradual heating to its final forming temperature and a rather sudden air quench to "freeze" the desired microtape shape in the filament.

A further aid to constant size microtape forming has been introduced recently as a result of the NASA Headquarters investigation (Contract No. NASW-1100). This consists of a flat rectangular hollow stainless steel chamber, large enough to accept the width of the preform, passing

vertically through the whole furnace from top to bottom. Attached to the bottom of the stainless steel chamber may be a water-cooled zone for air quenching the filament. The top of the chamber permits better draft seals to be installed between the chamber and the preform than is possible with an open furnace chamber. The stainless steel chamber then reradiates the heat from the non-metallic heating elements on either side of the chamber and at the same time minimizes the chimney effect draft whose turbulence can cause varying size filaments to be produced.

Early in the project, the original plan, to wind packages of microtape filament from which the tubes could then be filament wound, proved to be infeasible. The wide microtape with its large, flat contact area could not be unwound from a normally wound package and would have required the designing of an entirely different package and tensioning system, still with no guarantee of success. Therefore, in view of the relatively slow forming speeds of microtape, it was decided to go to direct and simultaneous filament forming and wet winding under the microtape forming furnace.

It was observed that even a dry, unbonded ribbon made up of a number of overlapped microtapes had surprisingly high tensile strength normal to the ribbon and in the plane of the microtape. From this behavior, it seemed possible that a hoop wound cylinder made from many layers of edge-to-edge microtape might have sufficient axial tensile strength to provide the requisite biaxial strength in a complete cylinder, circumventing the necessity for any axial wound microtape.

Microtape Cross-Sectional Shape and Size

During the early portion of this project an effort was made to produce microtape with a very high aspect ratio up to 100:1 width-to-thickness and very low thickness, even less than 10 microns. It was found that the original furnace design with its short flat horizontal hot zone produced microtape which was very thin across most of its width but which was much thicker on its edges. It was this problem that brought about the improved furnace design described above, which produced virtually flat microtape. Even so, some loss in aspect ratio from that of the preform was found to take place in forming flat microtape. Furthermore, during the later winding studies it became clear that a larger microtape than the 0.015 to 0.020" wide by 0.0005" thick tape was desirable to have a sufficion "curb" to abutt adjacent tapes against one another. A tape 0.060" wide and 0.0015" thick was used in the final stages of the work and in winding the cylinders submitted to NASA for test. Although a 50:1 aspect ratio was a target, 40:1 was found to be more reasonable and capable of being achieved reliably.

Microtape Placement

During early phases of the work, literally hundreds of trials were with the microtape drawing unit. Most of these were made dry to ascertain tape size and placement. Many trials were also required to work out the best guide design and material. It was learned that a microtape with the following characteristics can reliably be made:

Dimensions:

Width - .0162" $\pm$ .0005" over a period of 20 min.
$\pm$ .0001" for one min.

Thickness - .00044"

Width-to-thickness ratio - 37:1

Flatness - Only 6×10^{-5}" thicker at ends of 4.4×10^{-4}" thick tape

Strength:

Based on a loop test, the strength of this microtape made from window type glass is greater than 75,000 psi at room temperature.

Considering the above dimensions, let us now examine the requirements for placement in a structure to achieve 90% by volume of glass. Estimating 3% of the volume is taken up by the resin between the flat surfaces of the microtape, this leaves 7% which can be utilized in spacing the tapes beside one another. A gap of 7% of the overall width of the microtape is .0011". This emphasizes two things: first, the width of the tape must be kept more constant than was measured over a twenty minute period; secondly, the tape must be placed more accurately than $\pm$ .00055" from one tape to the next adjacent tape. Wider constant width tape would provide a larger tolerance of tape placement.

It was direct confrontation with such small placement dimensions and tolerances that lead us eventually to work with larger filament.

The furnace design and operating technique were further refined. To minimize the chimney or suction effect of the coupling agent chamber on the drawing furnace, it was eliminated and the expedient was used of adding the coupling agent directly to the resin. These moves have led to the production of the more uniform tape size. From the knowledge gained about the furnace design and operation, a still better furnace could be built which

would yield microtape whose width should vary even less and the same furnace should be capable of making wider microtape.

Much of the work during the latter part of Phase I was directed at examining the tape placement. As mentioned earlier, the guide material and design is closely related to this problem. A dense graphite guide with a narrow flat-bottomed groove has proved satisfactory after all. What has become apparent is that we have exceeded the capabilities of our mechanical traverse mechanism, the device that places the tape onto the mandrel beside its neighbor.

The winder used during Phase I, built at considerable expense to DeBell & Richardson, is a custom-built unit made to provide level winding over a width of from 1/4 to 2" at a wide variation in speed. During your program, it was found necessary to go to a special motor and speed control to maintain the selected speed more constant. The oscillating linear motion is produced by a coarse endless thread whose follower cam is connected through a ratio arm to the ball-bushing mounted winder table. The special well regulated variable speed motor and mandrel were mounted on the table. Since the table travels at a speed of only 4" per minute for a 16 mil tape being formed at 300' per minute, it was possible to counterbalance the table with a cable and weight to minimize backlash in the mechanical linkage.

Within the instrumentation at DeBell & Richardson, no way was found to directly measure how constant is the speed of the winder table. However, the multitude of microscopic examinations of wound structures, either transferred with transparent adhesive tape or cemented in resin, has shown too random a spacing within adjacent tapes of any one layer to obtain the objective of 90% glass. Observations further show that the random filament spacing is probably not the fault of the guid material or design. This leaves only erratic motion of the winder table as the most obvious cause of this random spacing. Working with multiple filaments or not, the same side-by-side filament placement problem must be solved. A solution must also be found even when winding in only one direction.

On the basis of the promising results but the need for still further placement improvement, a commercial textile spooler, Foster #607, was acquired. This was obtained with specifications that would facilitate modifying to a microtape winder.

Features include:

(a) one variable speed motor coupled directly through a right angle drive to both traversing and winding mechanisms,

(b) traverse advance, to match tape width, fixed by gear ratio chosen,

(c) traverse accomplished by a blade following the root of first one and then the other of two contrarotating running threads,

(d) length of traverse adjustable by means of replaceable fixed cams.

The modified spooler was first set up mounted on ball bushings operating on 3/4" diameter hardened shafts, in such a way that the guide could remain fixed while the whole mechanism: spooler, motor, right angle drive and all, would traverse. The blade follower proved inadequate to do this and still maintain the requisite traverse precision.

Later the spooler was inverted, anchoring the frame and motor, and a traversing guide support was provided. The rigidity and smoothness of operation was improved by replacing some of the 1/2" diameter shafts in the spooler mechanism with 3/4" diameter shafts and bearings where indicated. This arrangement has proved quite satisfactory and is the one on which the Phase IA research specimens were produced.

The original plan was to apply coupling agents in a column beneath the forming furnace in view of the good results obtained in this laboratory by vapor deposition of the coupling agents. Unfortunately, the effect of the tall vapor column chimney upon the forming furnace caused much unwanted turbulence and consequent variation in fiber size. The other disadvantage of a vapor column was that it was in the way. The chamber would have complicated the hundreds of experimental starts which have been typical of the operation to date. At the present time, the coupling agent is incorporated in the resin mix which is applied to the microtape. Now that the technique incorporates a chamber within the furnace, which lends itself to tighter draft control, it is possible that a vapor deposition technique could be used more satisfactorily.

Dynamic Tape Width Monitoring

One of the biggest steps forward in the mechanics of controlling the precision of tape size and, therefore, facilitating precise, close placement, was the introduction of an optical comparator type unit located immediately beneath the microtape forming furnace.

Figure 6 shows the unit, actually an 8 mm movie projector, in place with a dummy microtape threaded through the guides for illustrative purposes. Glass microtape is much smaller and of lower visibility. The microtape passes through the film gate and its image is projected onto a screen about five feet away. Figure 7 shows an actual microtape filament image on this screen. A metric scale shows this image of a .023" wide microtape

(typical is about .016") to be about 31 mm wide. It is possible to read the width to about two percent. The optical monitor is very useful to maintain a width below a set limit. Microtape cannot be successfully wound if its width exceeds the traverse spacing established by the precision winder.

The projector has become, almost immediately, an indispensable tool. First, it has the important job of width monitoring and permits accurate adjustment of width while running by means of the vernier control on the winder motor.

It seems quite possible this function could be automated although it is not contemplated at this time.

An unexpected bonus has been the contribution it has made in studying the effects of the condition of the surface and edges of the preform. Sheet glass, as mentioned earlier, during storage in contact with paper separators in a moderately humid location over a period of several months, develops a surface scum. If such a sheet of glass is used as a preform with no pre-cleaning, not only is the microtape product markedly weaker but also its width varies erratically while being formed. Simply by vigorous cleaning with a household glass cleaner, this effect can be brought under control as was observed with the optical comparator.

Furthermore, if a sheet of glass is cut poorly with too much pressure on the cutter, causing excessive chipping and an unusually ragged edge, the roughness can easily be seen in the width variations of the microtape. Later a new glass cutting technique is described which appears to overcome this problem. Also, as could be predicted, the incomplete removal of any etching residue from the cut edges of a preform, where it is more tenacious, yields microtape whose width will momentarily increase from time to time. When all factors are under control, the image of the microtape is so stationary, it appears as though the tape is not moving.

Movies of the fluttering image have been taken and comparative shots will be made of normal or desirable operation.

Glass Flow Studies

Another new technique has been considered because of its help in observing the flow of glass from the preform into the microtape. Before inserting a preform into the furnace, a heat resistant ceramic decalcomania in the form of a grid is applied to the preform. As can be seen in Figure 8, the flow pattern is rather evident. In spite of the fact that a flat, rectangular microtape was produced, the glass from the center of the preform draws down first. Therefore, the final forming

of the microtape must entail the thicker edges coming together to re-thicken the center to make a flat microtape.

This technique should be investigated in more detail. A finer grid is indicated and this could be obtained by shifting one decal with respect to the other to make an 1/8" grid instead of the 1/4" grid shown in Figure 8.

Microtape Strength Studies

One of the tasks specified for this project was the determination of microtape single filament strengths as well as a comparison with strengths of round fibers made by other means such as from a traditional bushing and attenuated from a rod.

It is not generally appreciated how complicated and exacting the testing of a glass filament can be. An unprotected filament loses strength with time. Therefore, tests must be run shortly after forming to observe maximum values. All brittle materials are very sensitive to any bending movement during a tensile test causing artifically low values so careful attention to gripping means is essential. The surface condition of a glass fiber affects its strength; a finger print can markedly reduce the strength of a glass fiber.

In preform attenuation drawing of shaped filaments, the condition of the surface of the preform affects the strength of the filament. As proposed, acid etching was utilized to prepare the surface of a preform for higher strength filament forming. This has entailed precleaning, etching with a suitable acid composition and concentration, rinsing and drying with evaporative solvent.

This appears to be straightforward. However, a number of hazards still exist. Dust in the air of the laboratory can collect on the preform surface. Dust or refractory particles from the interior of the furnace can be sources of stress concentrations. Devitrification, although not the major problem with window glass that it would be with borosilicate (Pyrex) or E glass, can markedly reduce the strength of a glass filament.

In an effort to run some unetched controls, it was found that sheet glass which had been stored in the normal paper packing had developed a faint scum which produced unusually low strength microtape. Only by careful cleaning of the glass with a commercial glass cleaner prior to drawing a fiber was the strength substantially increased. There is much literature on the strengthening of glass through acid etching or polishing. A purely qualitative demonstration brings home how effective

acid etching can be. After one end of a narrow strip of sheet glass is etched, it is remarkable how much more effort is required to manually break the etched end than is needed to break the unetched end. This is even more surprising in view of the fact that the etched end is smaller in cross sectional area than the unetched.

The literature does not suggest one particular acid bath composition or concentration as being ideal. Our investigation also showed there is no one clear cut answer. Hydrofluoric acid by itself in various concentrations leaves a tenacious reaction product on the surface of the glass which must be removed prior to filament drawing to take advantage of the strengthening. The precipitate, if left on the surface, can actually weaken the fiber drawn from a coated preform. A mixture of hydrofluoric, sulphuric and phosphoric acids gave good results and left a deposit that was much easier to remove. Agitation during etching may or may not assist in making stronger preforms although it can keep the deposit from clinging to the glass surface. A recommended procedure is continual scrubbing of the preform during etching. More will be done in this area although hydrofluoric acid is an extremely hazardous chemical.

A careful procedure must be worked out from pre-cleaning through etching and filament forming and including the care required in handling the filament prior to and during test or winding.

Strength Tests Summary - Tensile

1. One Hole Bushing - Window Glass

Bushing Temperature	2250°F
Fiber Diameter	.00030"
Strength	300,000 psi
Bushing Temperature	2300°F
Fiber Diameter	.00036"
Strength	343,000 psi
	310,000 psi
	288,000 psi (tested after 2 hrs.)

Bushing Temperature	2400°F Nitrogen jet across tip	
Fiber Diameter	.00036"	
Strength	360,000 psi	194,000 psi
	420,000	334,000
	280,000	460,000
	345,000	388,000
	366,000	216,000
	216,000	

Maximum - 460,000 psi
Average - 326,000 psi

2. Round fiber From Narrow Window Glass Strips

Diameter	.00042"
Strength	64,000 psi
	80,000

No Guides:

Diameter - .00042"	Diameter - .00035"
Strength - 148,000 psi	Strength - 156,000 psi
80,000	145,000
68,000	133,000
	111,000
	111,000
	89,000

3. Round Fiber From Window Glass Strip - Improved Furnace

Diameter - .00042"	
Strength - 272,000 psi	384,000 psi
304,000	400,000
320,000	320,000
416,000	320,000

Maximum - 416,000 psi
Average - 342,000 psi

4. Strength Comparison of 7/8" Wide, Steel Wheel Cut Strips Prepared as Follows:

A - Untreated - cut, cleaned, drawn
B - Acid etched
C - Fire polished edges

	A	B	C
	170,000 psi	190,000 psi	140,000 psi
	120,000	170,000	120,000
	165,000	200,000	130,000
	150,000	140,000	120,000
	120,000	120,000	115,000
	110,000	125,000	150,000
	145,000	185,000	215,000
Maximum -	170,000	200,000	215,000
Average -	140,000	160,000	141,000

5. Microtape From 3" Wide Wire Cut Preform

Filament - .015" x .00049"

Strength -	120,000 psi	155,000 psi
	103,000	118,000
	112,000	92,000

Maximum - 155,000 psi
Average - 117,000 psi

6. Microtape From 3" Wide Wire Cut Preform Then Acid Etched

Filament - .0144" x .00042"

Strength -	216,000 psi	178,000 psi
	178,000	222,000
	148,000	222,000
	133,000	

Maximum - 222,000 psi
Average - 185,000 psi

The summary of strength measurements above is only representative of a larger number of measurements that have been made during this study. Because of the large number of effects, mentioned earlier, it is difficult to completely control a long series of glass fiber strength tests and have truly comparative values.

The first series of tests listed above (1) show how bushing operating conditions can affect the strength of the product. The final values probably

approach optimum drawing conditions for a one hole bushing.

Next (2) are shown some of the results of efforts to make round fiber from a strip of window glass. With the single, small round filament, there is evidence that even clean graphite guides can lower its strength.

Following (3) are the results that show how drawing conditions can affect the strength results. These values, obtained with the improved furnace incorporating a top cooler projecting down into the hot zone, are probably near optimum for a round fiber drawn from a rectangular rod.

The next series of tests (4) show rather inconclusive data which seem to show a slight advantage for acid etching. Even less clear is the potential of fire polishing the edges of a sheet of glass with more spread to the values but little improvement overall. However, it was the work with preheating for edge fire polishing that led to the hot wire cutting.

The group of values (5) for a 3" wide, hot wire cut preform are disappointingly low and may indicate some overshadowing effect such as improperly cleaned surfaces.

The final data (6) contain encouraging results obtained recently from one hot wire cut 3" preform which was then acid etched. It is proposed to use this technique to produce microtape for winding into test specimens.

Most of the filament strength measurements were made on the simple tensile tester designed and assembled from components at this laboratory using the appropriate size minature dynamometers. Figure 9 shows this unit. To minimize the effect of the rotary motion of the test arm, the dynamometers are driven slowly in a clockwise direction at a speed to achieve a test time of about 6 seconds. This is equivalent to the test time achieved on the Instron tester at .05"/min. crosshead travel. The other end of the filament is anchored to a stationary member. The fine wires are connected to heating elements used to melt sealing wax for fastening the filament in place. This is satisfactory for round glass filaments. Microtape is fastened to flat grips by means of cellulose adhesive tape or Eastman 910 cement. In the foreground, there are two dynamometers of different ranges which can be fastened into the same rotating holder. The final drive for the dynamometers is through a magnetic clutch so any overrun of the dynamometer can be stopped at the end of the test. The holder can then be repositioned for inserting the next specimen. It is felt that this tensile tester is suitable for rapid sorting tests and probably achieves test values within 10% of true tensile loads. Tests can be made within minutes of taking the samples.

At the suggestion of DeBell & Richardson, Inc., the program was enlarged to include the fabrication of some smaller diameter (2-3/8") thin-walled 0.040" hoop-wound microtape tubes. At Lewis Research Center, end caps were cemented onto short lengths of the tubes and they were tested to failure from internal pressure. This provides a specimen with no edge effect across the microtape filament ends. Damage from sample preparation will be eliminated. True values for the tensile strength of the cylinder wall in an axial direction are determined.

To date the vast majority of the microtape effort has been undertaken with window glass rather than Pyrex or E glass as originally proposed. A number of discussions have been held on this very subject with the technical monitor. Bushing-formed E glass has good tensile strength. Lime glass, such as window glass, has lower tensile strength and is subject to rapid degradation from moisture attack. However, E glass has a much shorter working range than window glass while Pyrex has a somewhat shorter range. The long working range of window glass is ideal for the preform attenuation process, especially with the long slow preheat found so desirable in the forming of high width-to-thickness ratio microtape.

Another shortcoming of both Pyrex and especially E glass is their tendency to devitrify or form microcrystals in the shaped filament drawing temperature range. These crystallites then become defects which drastically lower the filament strength. The bushing process can operate at a high enough temperature that devitrification is prevented, but the forming of shaped filaments from a preform requires a lower temperature with the glass spending enough time in the devitrification range to cause trouble. Window glass, on the other hand, resists devitrification for long periods of time.

Another distinct advantage of window glass is its availability in a number of thicknesses down to about 0.040". Pyrex is available in sheet form in a limited assortment of thicknesses and in limited area.

Therefor, window glass has proven to be the candidate material for this program. It is certainly a good model material. Should the outcome of the work look promising enough, a later project could be the development of a glass composition especially for this application which incorporates high filament strength, resistance to devitrification and long working range.

As a tool for examining incoming sheet glass and determining the improvement on acid polishing, some work was done using a microcrack and defect detection technique. Ernsberger (4) used this ion exchange method to study defects, microcracks and damage from polishing plate glass.

In the technique used, a piece of glass is immersed in a molten salt bath consisting of 40% lithium nitrate and 60% potassium nitrate. Through an ion exchange, the smaller lithium ion replaces the sodium ion in the surface layer which puts the surface under tension making it more subject to corrosion. Subsequent "development" in hydrofluoric acid etches out pits and cracks which had been present before immersing the glass in the salt bath as microcracks and defects. The interpretation is then made using low power microscopic examination. This technique would have to be refined still further to be able to use it as a quality control procedure in day to day microtape forming.

The hot wire sheet glass cutter was developed to cut preforms from larger sheets. The device has a straight resistance wire supported in a groove machined in the edge of a strip of asbestos-cement board. By holding the large sheet in intimate contact with the wire during rapid heating, it causes the sheet to crack by thermal shock. The whole sheet snaps into two pieces and leaves a smooth, surprisingly straight edge.

Figure 10 is a photograph showing the difference in the reflection from the edges of two strips of glass. Imagine you are looking at a black background through an almost fully opened jalousie window made with only two strips of glass. The light is positioned in such a way that both the front edge and the back edge of each strip reflects at a maximum. The labels are attached to the front edge of each strip. "A" is the strip cut with the hot wire while "B" was cut with a steel wheel cutter. Each strip is about the same width (7/8") and thickness (A = .050", B = .046").

The smooth fracture of the wire cut edge in "A" is apparent. The edge left from conventional glass cutting in "B" shows evidence of the irregularly rippled conchoidal fracture. Close examination reveals that the upper surface of "B" was scribed with the steel wheel and is an obvious source of further cracking. It is not possible to completely eradicate such roughness by acid etching.

Microtape is, in essence, a piece of flake glass of infinite length. The proposal to use resin-bonded microtape in the fabrication of containers for cryogenic liquids was made with a view toward combining the functions of liner and container in one composite. In NASA TM X-1193, Frischmuth (5) describes a liner fabricated from flake glass. This interesting liner material has the shortcoming that it requires bonding to the glass fiber cryogenic propellant tanks whereas microtape reinforcement makes a liner integral with the tank wall.

NASA TN D-3205 (6) and TN D-3206 (7) both indicate that a satisfactory reliable liner is not yet available. From the work on shaped glass fibers which has been done on NASW-672 and NASW-1100 for NASA Headquarters, it seems feasible to design a tank similar to the internally insulated tank described by Heidelberg (8), NASA TN D-3068, using hollow microtape and very hollow glass fibers as the core in the sandwich. Solid microtape could be used as the inner skin and any high strength glass fiber filament could be wound on the exterior.

Before the preliminary specimens were fabricated, a number of samples were wound on the new NASA Headquarters modified lathe winder. First, the equipment had to be checked out and optimum running conditions established.

A great deal of experience had been gained using the 0.0005" x 0.020" microtape so this size was used at the beginning of this winding study. Even with all the improved equipment and the accumulated fiber forming and precision winding experience, the small microtape was difficult to wind into 100% perfect specimens.

As an example, consider the 30" long, 0.040" wall, 2.3" I.D. tube. To wind a perfect specimen with the 0.020" wide microtape requires the drawing of a continuous length of filament over 11 miles long whose width does not vary more than ± 0.0005" and necessitates placement in the structure to the same tolerance. By microscope monitoring, the mandrel speed can be adjusted to compensate for gradual width changes; however, sudden fluctuations can be just as damaging.

Other investigators (9) have obtained good mechanical properties in filament wound structures with larger-than-standard diameter round filaments. A simple analysis showed that larger microtape filaments could be placed into a tighter overall structure; that is one with a lower percentage resin gap from edge-to-edge while still requiring less critical dimensional tolerances.

Various larger microtape filaments were drawn and a filament about 0.0015" thick and 0.060" wide was chosen as a size that could be made consistently to close dimensional tolerances with a good flat cross sectional shape and with sufficient strength to wind without breaking at the guides nor at the relatively small diameter mandrel. This larger filament would require only about 1-1/2 miles of microtape to make the same size tubular specimen. The specimen could have an improved structure even though the filament width tolerance was relaxed to ± 0.001" since the precision winder tolerances remain the same for any size filament.

A large number of tubes were wound for burst testing to obtain strength and modulus of elasticity data. As this series of specimens was made, under a combined Headquarters and Lewis effort, a proficiency was developed in making better structures by improved filament placement and a concurrent resin system evaluation was conducted. These data are reported in the Test Results section and are summarized in Table I while the resin systems are listed in Table II.

The data were developed on 2-3/8" diameter thin wall tubes wound from solid microtape. This specimen was chosen, after discussions with Lewis, to minimize end and edge effects in a specimen that was appropriate to make by precision filament winding with microtape.

In order to achieve even better filament placement than had been possible with the 0.020" wide by 0.0005" thick microtape, the larger tape described above was used, 0.060" wide by 0.0015" thick. Tubes 30" long were wound and these were cut into two or three equal lengths for testing.

Most of the tubes were tested by filling them with water and applying an internal pressure to them, pressurizing the water with compressed nitrogen. Steel caps were cemented to each end of the test section with an inlet in one of them for pressurizing.

Table I contains the data on the construction of the tubes (I-A) as well as the test results (I-B).

Ten different resin systems were used in an effort to find a system that would perform better in the microtape structure with its thin, approximately 1/2 micron, resin layers. It can be seen that the number of layers of microtape is approximately proportional to the thickness, which would be expected. The thickness shown has been measured through a micro-scope after the test was completed on a polished cross section cut from the wall of the tube near the point of failure. The measurement is made from the inside of the inner microtape layer to the outside of the outer glass layer. It includes all the glass as well as the thin interlaminar resin layers but it excludes the excess resin layer on the exterior surface of the tube as well as any small amount that may be present on the inside.

Most of the tests have been what is called "step burst" tests where the pressure was raised 100 psi and held for a minute after which readings were taken and the cycle is repeated; raised 100 psi, held one minute, then read. Procedures other than this are noted under the "Type Test" column.

The water pressures were read using various range Bourdon Tube pressure gauges. The maximum stress was calculated on the basis of the maximum pressure and the dimensions of the tube.

In order to obtain modulus data, small flat wire ribbons were spring loaded circumferentially and axially on the outside surface. A reference mark was scribed on adjacent ribbons and the position of the reference marks was read with a 6-power comparator to 0.001". These then generated stressstrain data for computing modulus.

All the tubes were pressurized with both ends free to move with respect to one another, except the first one which was tested first with the ends held together with tie bolts. As will be noted, while the ends were restrained failure did not occur up to 1500 psi internal pressure at which point the test was stopped although there was noticeable cracking. It was only after the ends were free to move that the tube burst. The lower modulus figures on the second test of this tube may be attributed to the resin cracking under the first pressurizing making the tube act softer upon retest.

The quick burst test gave the highest maximum stress. Since all the failures with the unrestrained tests appeared to be axial failures with fairly clean breaks, the circumferential maximum stress values are only of interest to see what levels were reached before failure occurred in the axial direction.

The modest stress levels at which failure has occurred has been interpreted as meaning that the proper resin system for the exceedingly thin, large area, resin layers has not been found. The requirements for a suitable resin could approach the properties proposed in the following hypothetical resin specification:

Requirements:

High elongation	$\geq$ 200, prefer $\geq$ 500%
Low cure shrinkage	$<$ 3%
High tensile strength	12,000 + psi
High shear strength	10,000 + psi

Long pot life
Viscosity of about 500 centipoises
Solventless system
Absolutely no volatiles during curing

The most striking feature of the test results is the unusually high modulus of elasticity demonstrated by the high glass content tubes. Soda-lime sheet glass with a modulus of elasticity of 10.0×10^6 psi was used as a preform stock from which the microtape was drawn. The resins have a modulus of less than 1×10^6. Instead of the material yielding values on the basis of mixtures, the resin seems to have lost any effect it might have on this structure to lower the modulus.

The spread of the modulus values is difficult to comprehend. The measurements were all made using the same direct reading technique. The results were all calculated very carefully using a graphical method or a least-squares analysis, and in some cases both. Since some circumferential values are higher than their axial counterparts and vice versa, no pattern exists here. Nor do they seem to be at all related to the resin systems.

It should be borne in mind that these elastic modulus values, as observed, are in a biaxial stress field. Using the effects of Poisson's ratio on parallel fibers in a biaxial field, such as the analysis in Appendix A, the average moduli would convert to 6.2×10^6 psi for a unidirectional tensile modulus in the axial direction and 8.8×10^6 psi in the hoop direction. These values are remarkably high compared to the lower values exhibited by normal filament wound composites, which behave more nearly the way mixtures would be predicted to behave.

A preliminary investigation into the torsional behavior of two 2-3/8" tubular samples was also undertaken. The table below compares microtape wound tubes with a test run on a steel tube of similar size as a control.

	Wall Thickness	Torsional Modulus psi x 10^6	Maximum Stress at Failure, psi x 10^3
Steel	.035	8.3	-
12/9 A	.022	2.7	19.2
12/9 B	.034	2.3	23.3

It would appear that microtape wound tubes could perform well as lightweight torque tubes.

Some early data obtained by compression testing of 2.6" diameter rings seemed to confirm the need for "edgeless", "endless" test specimens; namely, the long 2.3" diameter tubes.

In spite of the large overlap in the width direction if microtape in a circumferentially wound tube, internal pressure burst tests with unrestrained ends showed that the axial strength was low and the clean breaks around a circumference were evidence of axial failures.

In an effort to make a more balanced structure, some axial layers of microtape were incorporated in a few tubes (Table I, 12-22 AL, 12-22 AR). Although the failures were more ragged indicating a more nearly biaxial stress field, the results were no better, possibly due to the use of an improper resin system and the fact that these trial tubes were too thin, having only a 0.0153" wall compared with most of the tube test specimens with a 0.034" to 0.040" wall.

These cross wound tubes were made by first winding single layers of resin-wet microtape onto a plastic film transfer sheet on a large 9" diameter mandrel. A number of these single layers were cut off the mandrel and set aside. Then with the 2.3" diameter mandrel on the winder, after four circumferential layers had been applied, the machine was stopped while two single layers which had been made earlier on the 9" mandrel were carefully wrapped on top of the hoop winds with the microtape running in an axial direction. Finally, four more circumferential layers were wound on top to complete the structure.

Resin Layer Thickness Studies

Another task specified in this project concerned the measuring of resin layer thicknesses in a microtape wound tube. Resin layer measurements were made on a typical good quality structure by carefully polishing a cross section of a tube wall and examining it under the microscope. Viewing was done by transmitted light with an oil immersion lens giving a magnification of 970 diameters.

Four passes across a whole wall thickness were made with each resin layer measured. Figure 11 shows the resin thickness frequency distribution plots for each of these four traverses of a tube wall. There is a rather random spread of values as well as a thickness variation of 8 to 1. However, it should be borne in mind that all but one of the 80 measurements are less than 0.0001" and that one is only 0.00013".

The ultrathin layers would be expected to behave very differently from the much larger resin "rods" in among round glass fibers in a normal filament wound structure. Furthermore, the 8 to 1 variation in the bonding layer dimension could tax the resin properties still further.

Photomicrographs have been made of cross sections through the wall of a short end trimmed from each of the seven (7) 2.3" tubes sent to Lewis Research Center for testing. Figures 12 through 18, identified with their tube designations, at 100 diameters magnification, show the thin resin layers, the very flat 0.060" microtape and the somewhat variable edge-to-edge placement. The variations in surface finish in the glass areas and the different intensities are slightly different degrees of polish and lighting on the microscope and are not differences in the composites.

Filament Tension Study

A method was devised to measure filament tension during winding. Figure 19, with the various parts labeled, is a view along the 2.3" mandrel on the modified lathe winder. A white string has been used in this mock-up to show the route a microtape filament would normally take. Small graphite bobbins guide the filament onto the mandrel and two small tandem rubber wipers push the filament into place and smooth the resin preparatory to placing the microtape on the following revolution of the mandrel.

As a tensioning device, a third graphite bobbin was affixed to a small spring dynamometer attached to an adjustable platform on the floating guide holder. As can be seen from this arrangement, the actual tension in the filament at this point can be calculated from the load on the dynamometer and the angle the filament makes passing over the dynamometer guide. However, more tension of an unmeasurable increment is added at the lower two guides and still more is added by the action of the two wipers.

Typical running tension measurements were 5 or 6 grams, while by repositioning bobbin No. 1 values up to 9 grams were noted. Above this, the filament would not run because the increased tension at the measuring guide would change the angle, increasing the additional tension at bobbin No. 2. While running, the tension would vary less than 1/2 gram from the 5 gram setting through several complete 30" passes along the mandrel.

Since the amount of tension was found to be uniform and since no improvement in structure nor filament placement was noted, no further filament studies were made.

Lewis Research Center Test Results

Table II contains the results obtained when specimens of filament wound microtape made at DeBell & Richardson, Inc. were tested at Lewis Research Center.

The NOL rings, cut from a longer cylinder with a diamond wheel, show a predictable increase in tensile strength with reducing temperature. The strengths, ranging from 100 to 180 thousand psi, are only modest, typical of glass microtape as discussed earlier, the result of relatively cold filament forming.

The transverse shear tests are the results obtained by pulling micro-tensile specimens which were cut in the axial direction of the cylinders. Thus the result is a measure of the shear strength developed as the edges of the microtape overlap one another. These values are low, in the range of one half to one third the level of values obtained in burst testing of cylinders. It is felt that the tensile specimens cannot be ground out of the thin walled high glass content cylinders without weakening them.

The results obtained at Lewis Research Center, burst testing 2-3/8" thin walled tubes at room temperature, are shown in Table III. These results agree well with those obtained during similiar tests at DeBell & Richardson, Inc. and shown in Table I. Lewis also made straight axial tensile tests of some cylinders getting results, listed as tensile tests, in good agreement with the burst tests. The micro tensile tests reported in Table II should yield values as high as these. Most of the low temperature values are not higher which may indicate that just the act of taking the specimens to low temperature has caused considerable damage with the thin resin layers of low elongation resin and a big differential in coefficient of expansion.

Lewis also measured the pressure drop with time of several microtape wound cylinders and compared them with ordinary filament wound tube, at various pressures and in one case at LN_2 temperature. The latter was so self destroying as to lose over 400 psi immediately.

A microtape tube pressurized to 1000 psi with N_2 gas dropped 260 psi in 30 minutes while an unlined normal FRP tube dropped 340 psi in 20 minutes when pressurized to only 800 pounds.

At levels of 400 and 600 psi microtape tubes appeared to approach static pressure drops of 30 and 50 psi respectively.

CONCLUSIONS

Through refinement of DeBell & Richardson's preform attenuation technique for drawing shaped glass fibers, it has been demonstrated that a uniform, high width-to-thickness ratio glass microtape can be drawn from a sheet of soda-lime glass and can be filament-wound into a high glass content thin-walled cylinder using epoxy resin binders. In the course of this project, a number of intermediate objectives were reached. These comprised the producing of samples meeting some particular specifications and the development of techniques to assist in reaching these goals. The following list includes most of these:

1. Delivered microtape NOL rings and microtensile specimens as well as 2-3/8" and 5-3/4" diameter cylinders to NASA Lewis Research Center.

2. Achieved over 85% glass content by volume microtape filament wound structures.

3. Evolved acid polishing technique for window glass which more than doubled the strength of filaments made therefrom.

4. Applied ion exchange technique to sheet glass both before and after polishing to ascertain quality and characterize microscopic and submicroscopic surface flaws.

5. Developed hot wire cutter to eliminate ragged cut edge produced by common cutters such as steel or carbide wheels or diamond tips.

6. Refined preform attenuation technique to the degree that microtape can be drawn continuously to a prescribed size and shape.

7. Developed a precision winding method capable of virtually edge-to-edge winding of single microtape.

Research specimens with high quality microtape filament windings have been supplied to Lewis. The mechanical problems of fabricating tubular specimens from resin-wet glass microtape have been solved to a large extent.

Burst tests at DeBell & Richardson, Inc. as well as tests at Lewis seem to indicate that the full strength of the glass is not being utilized.

The resin layers are, typically, 1 micron thick with many layers only 1/4 of a micron thick.

Filament tension measurements made during winding indicate that the tension is very constant during winding. Varying this tension somewhat did not appear to change structure.

All the evidence seems to point to resin failures limiting the performance of microtape filament wound tubes. A preliminary cross winding trial yielded no additional axial strength. Prior to failure in the burst tests, prominent, audible and visual cracks or crazed areas appear. Although no positive evidence has been developed, microscopic examination tends to confirm that the crazing is in the ultrathin resin layers.

Two limitations of the resin-coupling agent systems may be the cause of the premature, lower-than-glass-strength failures. The more likely reason is that the resin system has insufficient elongation. Such thin resin layers may well require more nearly an adhesive function than a bonding; casting function. Another possibility is that the coupling agent introduced in the resin is not performing as efficiently as is required in this stringent application.

The structure developed on this project for Lewis Research Center did not perform satisfactorily as a cryogenic liquid container. Substantially more development is indicated before it might be useful in this area. However, it seems highly probable that this transparent, low permeability, high modulus, high glass content structure could be suitable for many other applications even at its present stage of development.

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NASA CR-517 "Precision Winding of Cylindrical Composites with Shaped Glass Filaments" by Richard S. Humphrey, August, 1966. This is one of two co-issuing final reports on NASw-1100, a continuation of NASw-672 mentioned in Reference 1. The other final report is NASA CR-518 "Initiation of Failure Mechanisms in Glass-Resin Composites" by William J. Eakins.

FIGURES

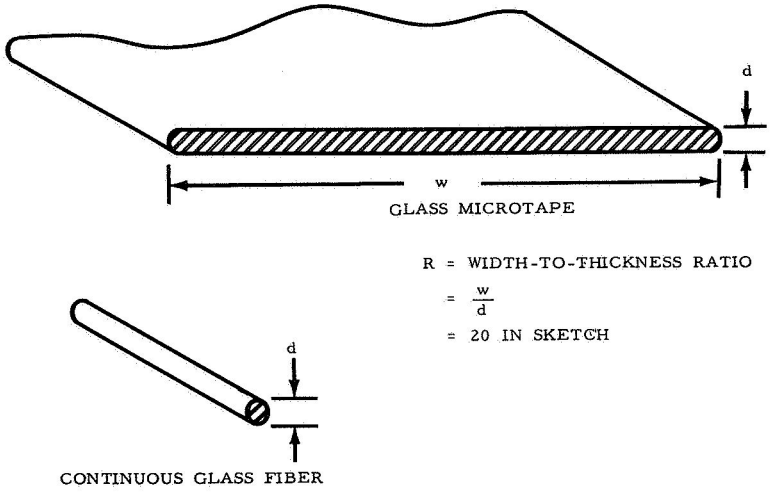


Figure 1. Comparison of Glass Microtape With Glass Fiber

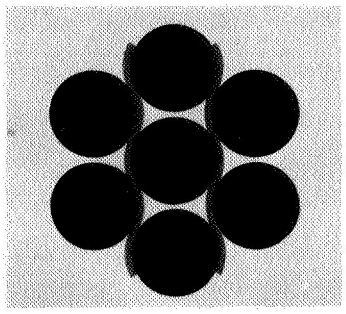


Figure 2. Diagram of Leakage Path Through Round Filaments Hexagonal Packing

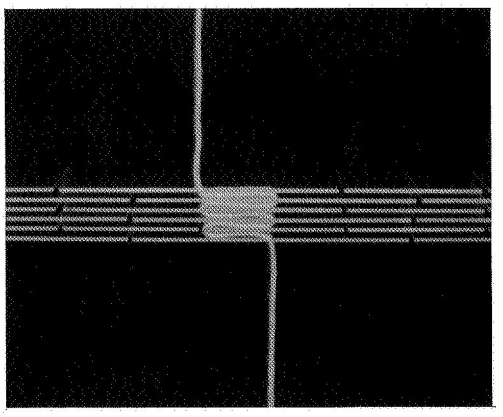


Figure 3. Model of Microtape Composite Cross Section With Perfect Overlap Showing Leakage Path.

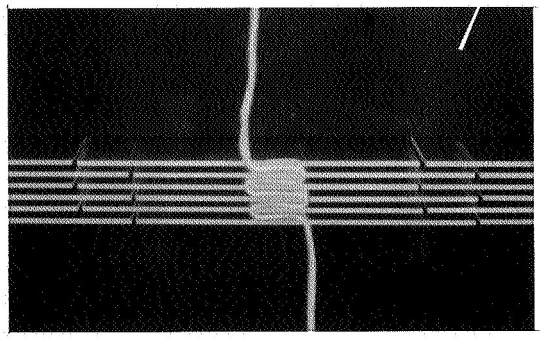


Figure 4. Similiar Model But With One-Third Overlap Showing Shorter Leakage Path.

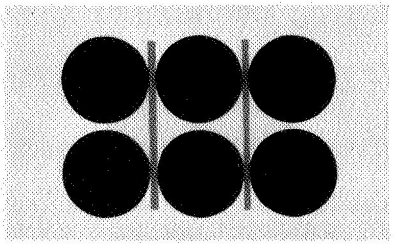


Figure 5. Diagram of Leakage Path Through Round Filaments - Square Packing

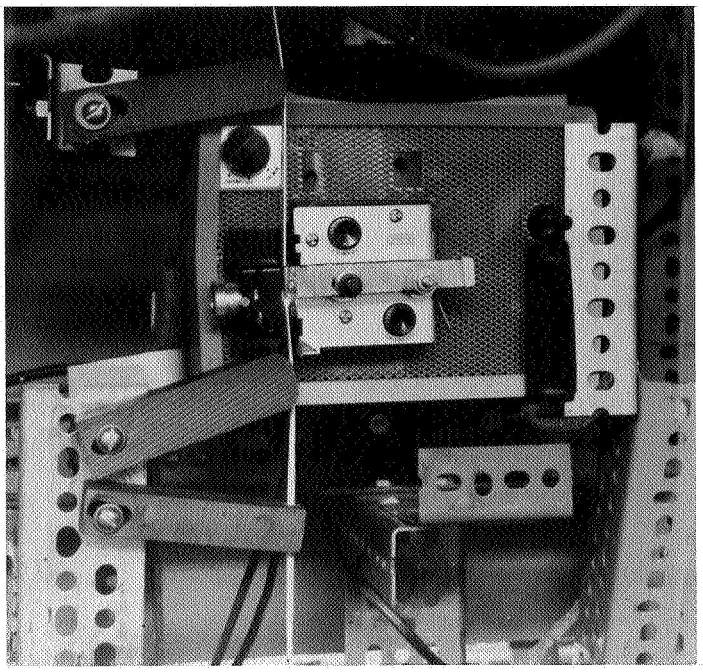


Figure 6. Optical Comparator.

Movie projector (8mm) mounted beneath furnace to project enlarged image of microtape onto screen for monitoring width. In addition to graphite guides above and below projector, note small, adjustable guide added to film gate area to keep microtape (a white tape for this photograph) in focal plane.

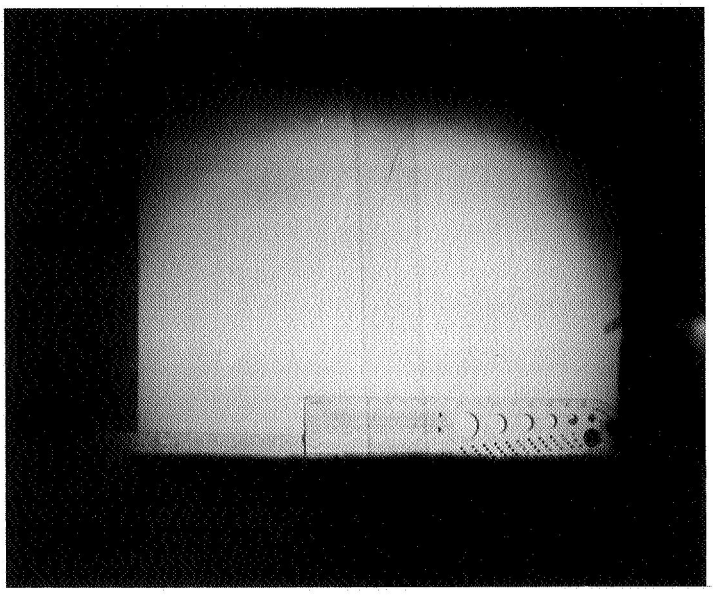


Figure 7. Image of microtape on screen. Microtape - .023"; image - 31 mm.

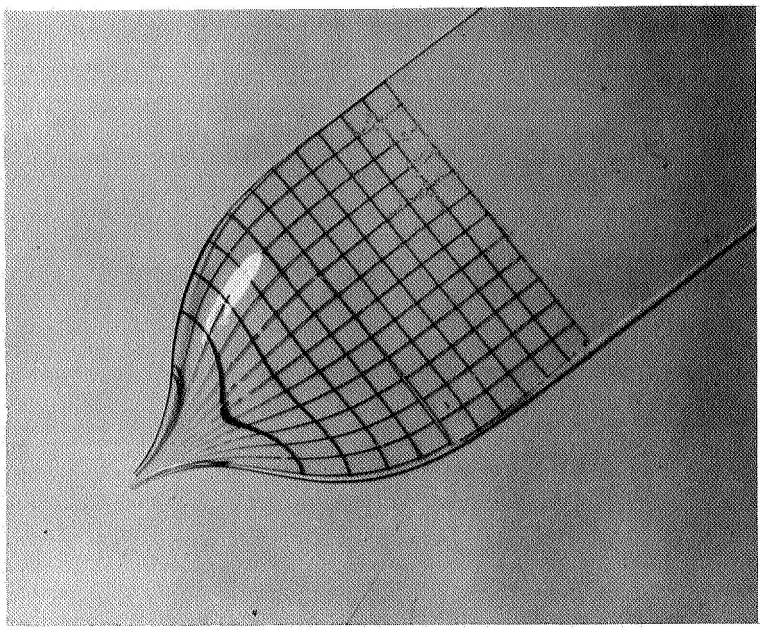


Figure 8. Preform removed from furnace while drawing microtape. Heat resistant decalcomania grid shows glass flow at time of removal.

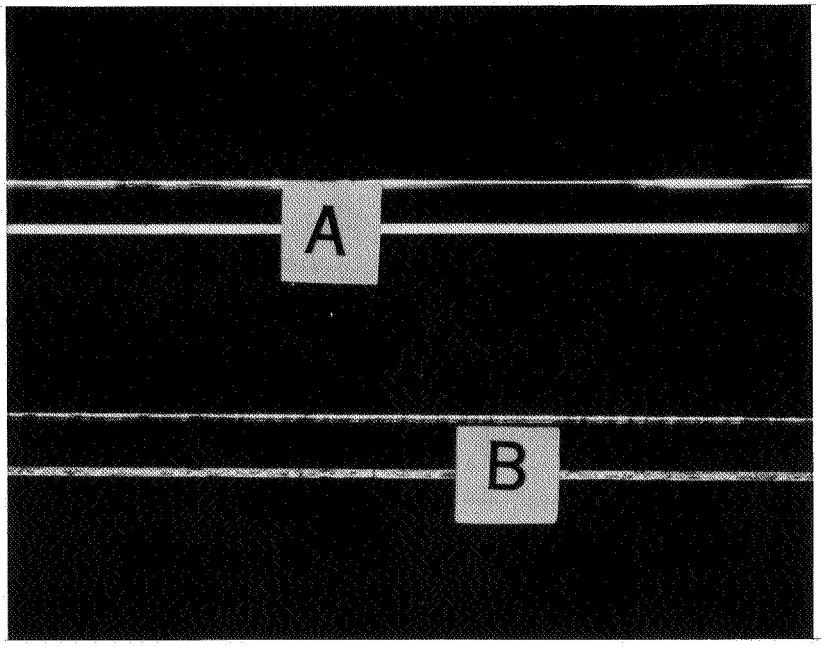


Figure 10. Two strips of sheet glass slanted to reflect light from both front and rear edges. No light is reflected from faces of strips.

A - hot wire cut lantern slide strip of glass

B - steel wheel cut lantern slide strip of glass

Note smooth cut at "A" and irregular cut at "B".

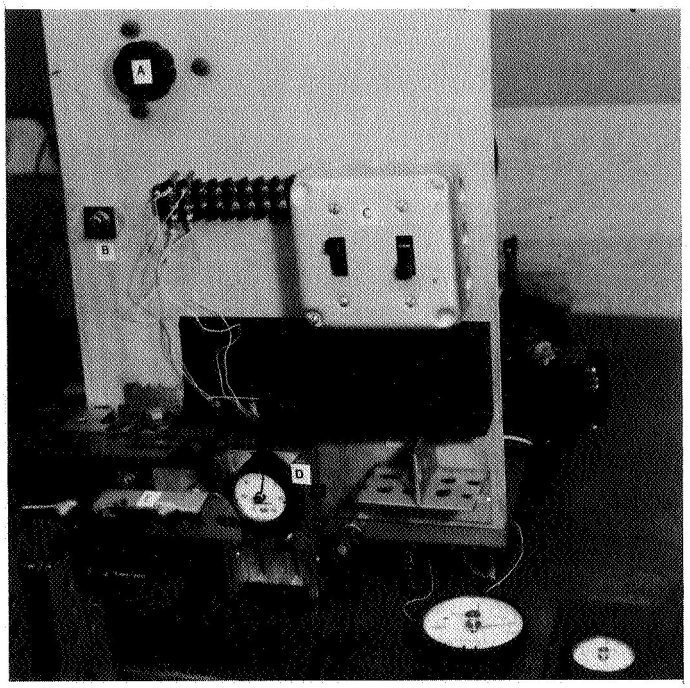


Figure 9. Filament Tensile Strength Tester

A. Variable transformer for heat control of sealing wax grips.

B. Momentary push button to connect power to sealing wax heaters.

C. Switches for magnetic clutch and drive motor.

D. Rotating holder for dynamometer (shown at rest against adjustable stop in "start" position).

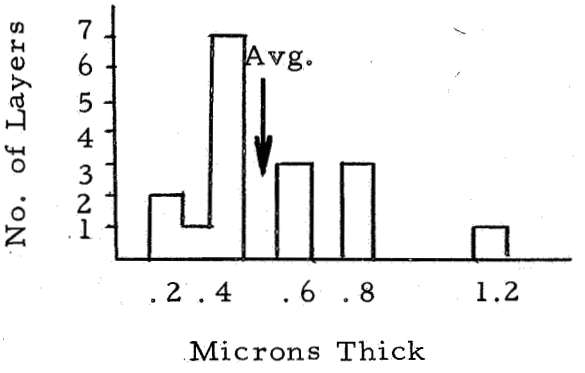
E. A hinged, two-tine fork for positioning test filament against sealing wax.

The dynamometer holder (D) rotates in a clockwise direction around the axis of the dynamometer torque arm. This in turn applies a known tension to the filament through the torque arm while the other, right end of the filament is anchored stationary. A dummy, white filament is shown in place and the ends of it can be seen still in the mounting fork (E) swung closer to the viewer and somewhat out of focus. The tensile failure load is marked by a lazy pointer on the dynamometer.

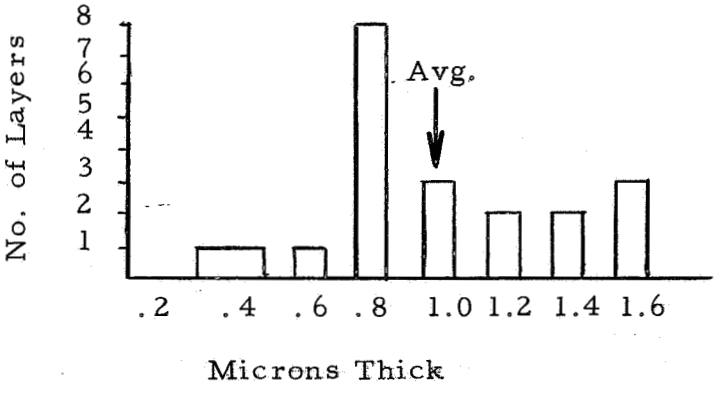
FIGURE 11

Resin Thickness Frequency Distribution

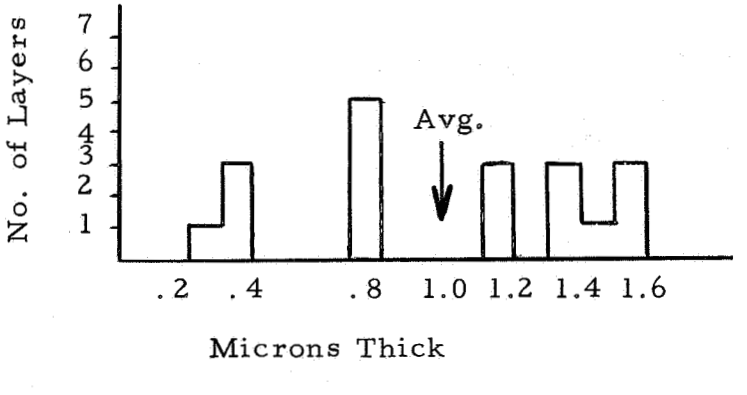
Traverse 1



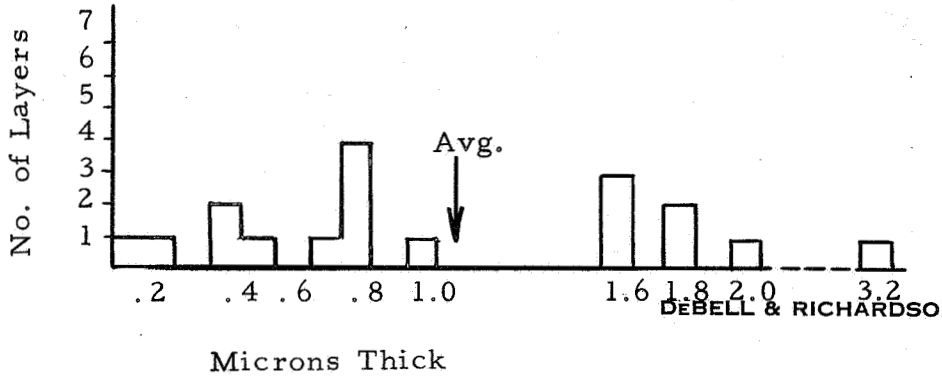
Traverse 2



Traverse 3



Traverse 4



Cross Sections Through 2.3" Research Specimens

100 X

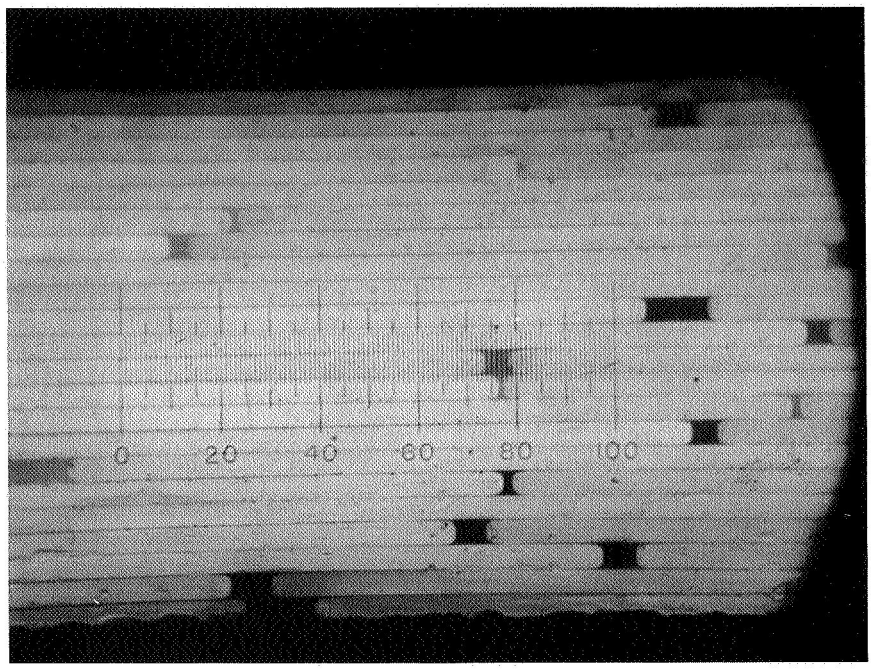


Figure 12 - Tube 1-24-66

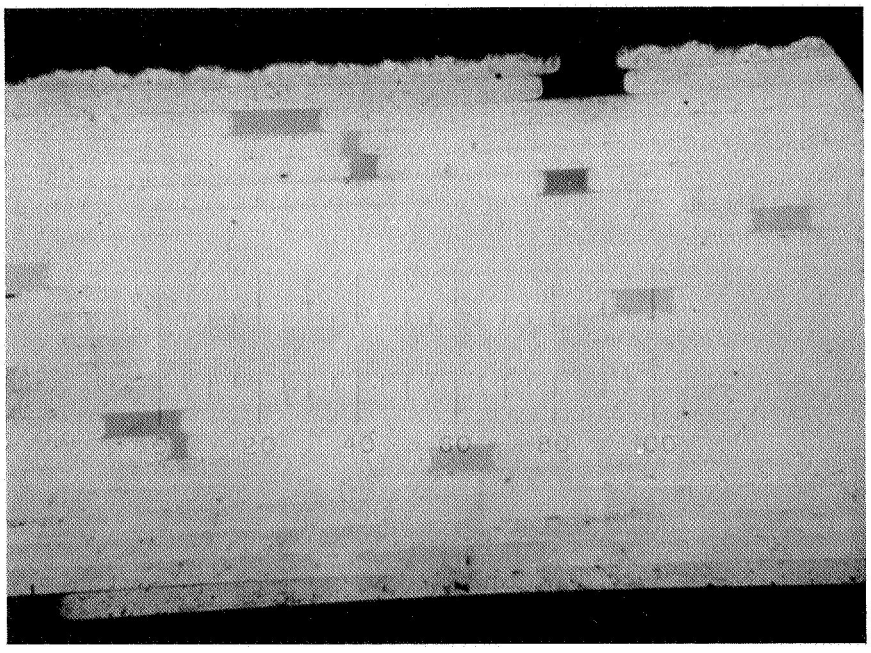


Figure 13 - Tube 1-25-66 A

Cross Sections Through 2.3" Research Specimens

100 X

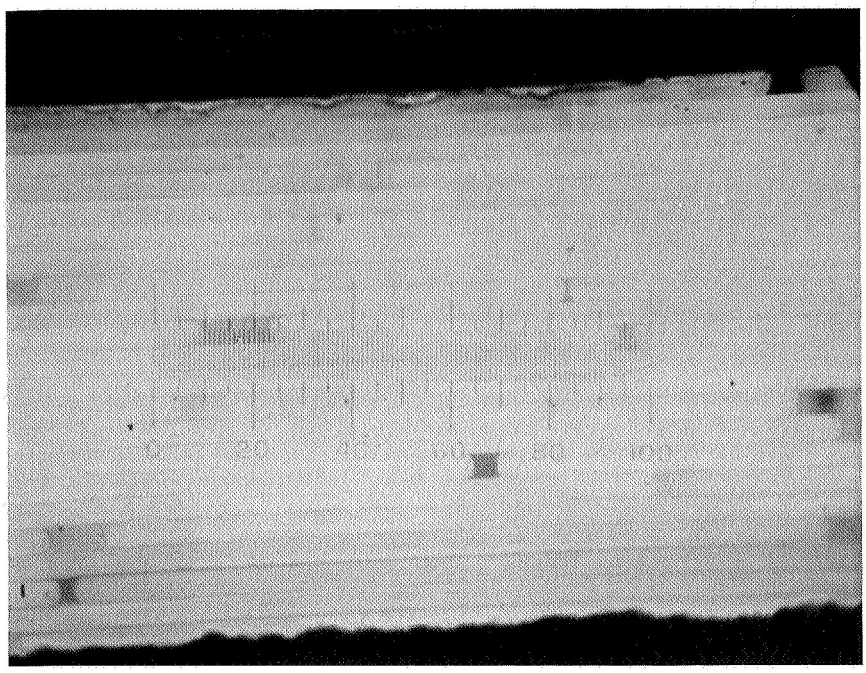


Figure 14 - Tube 1-25-66 B

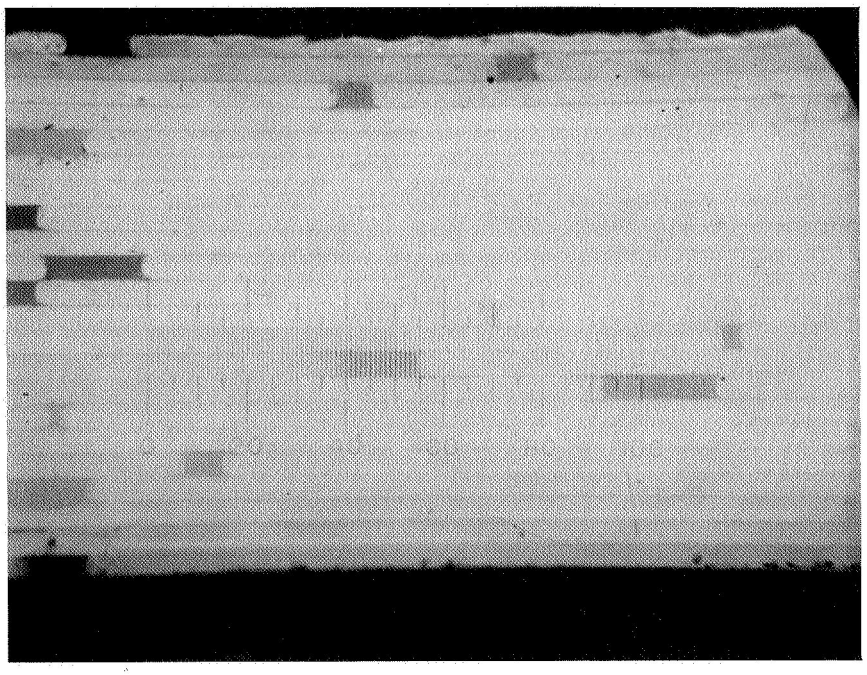


Figure 15 - Tube 1-26-66 A

Cross Sections Through 2.3" Research Specimens

100 X

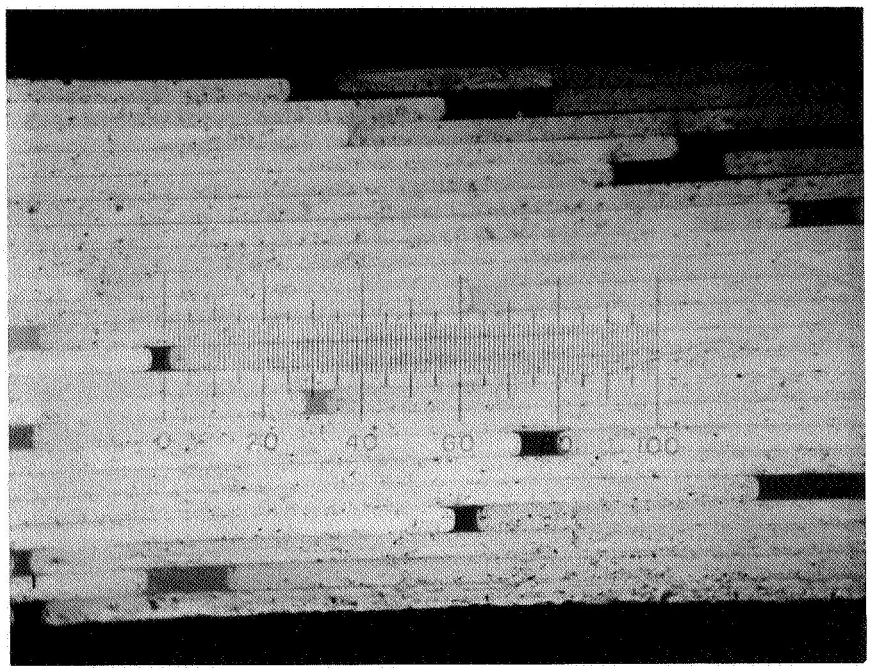


Figure 16 - Tube 1-26-66 B

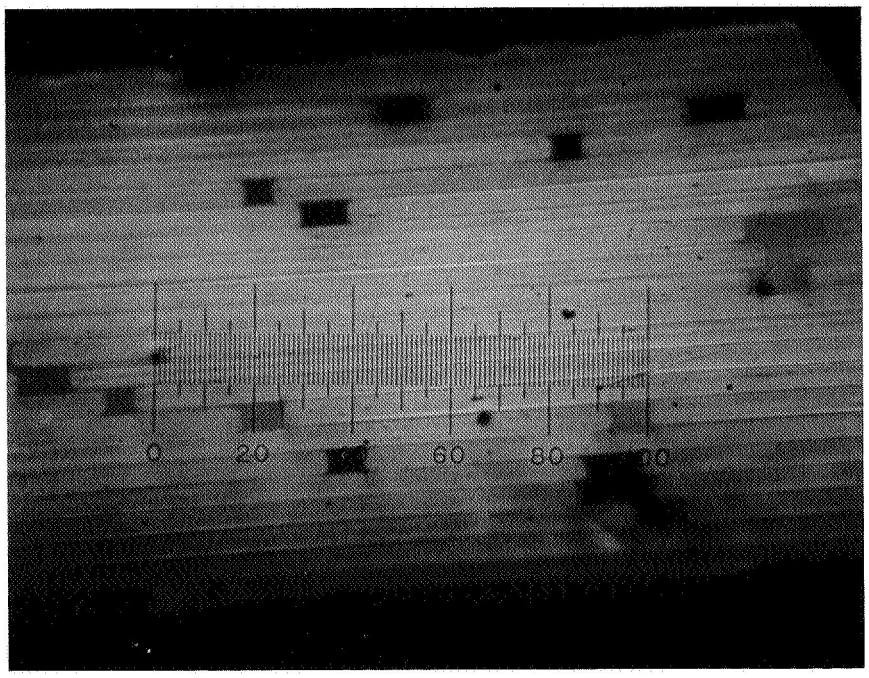


Figure 17 - Tube 1-27-66 A

Cross Sections Through 2.3" Research Specimens

100 X

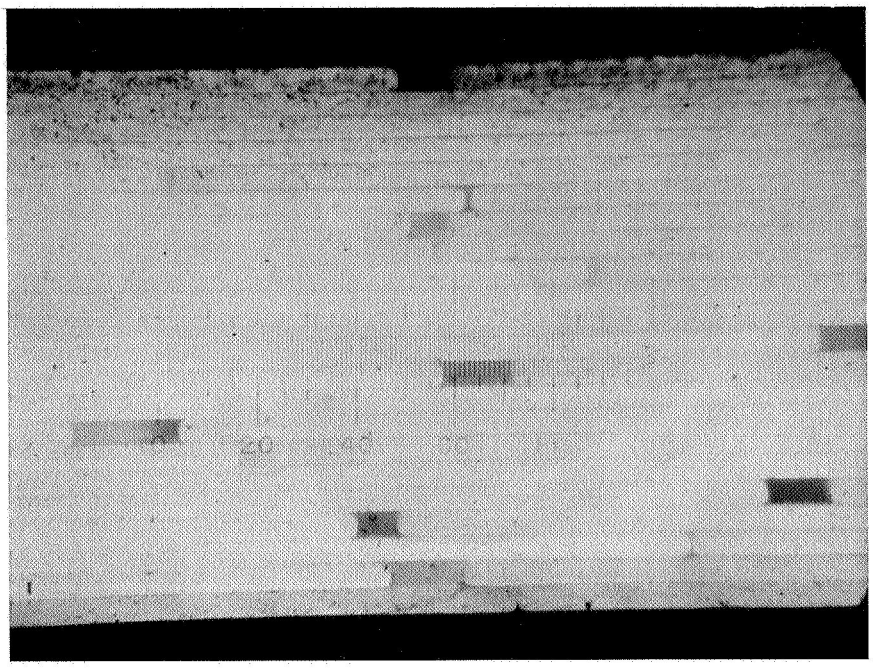


Figure 18 - Tube 1-27-66 B

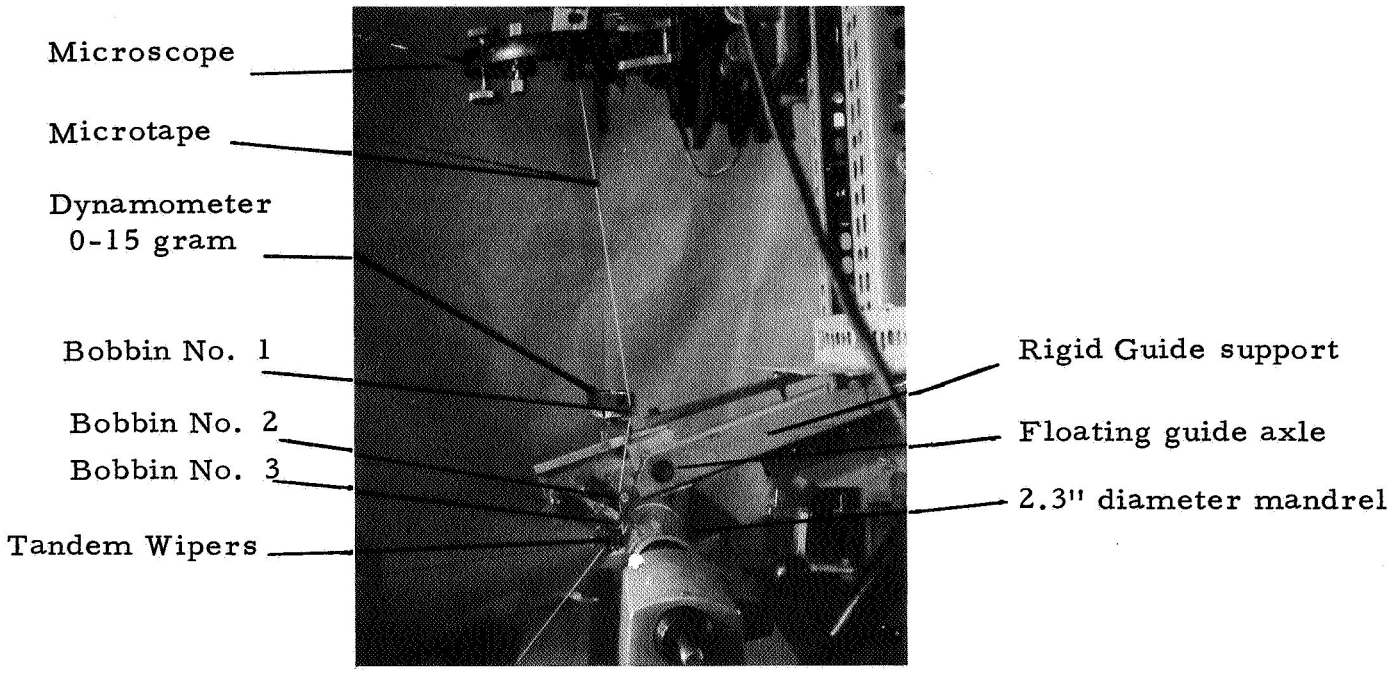


Figure 19

End View Modified Lathe Winder Showing Filament

Tension Measuring Arrangement

TABLE I

Internal Pressure Tests - 2-3/8" Diameter Microtape Tubes

I-A - Construction and Type of Test

Tube Designation	Resin System (Table II)	Layers of Microtape	Thickness, in.	Type Test
10-21 R	1	19	0.038	Step, restrained end, no burst
10-21 R	1	19	0.038	Same specimen, step burst
10-20	2	17	0.034	Step
10-26 L	3	22	0.037	Step
10-26 R	3	22	0.037	Quick burst, 0.78 min.
10-27 R	3	22	0.037	Step
11-5 R	3a	19	0.034	Step
11-5 L	3a	19	0.034	Fatigue
11-8 L	1	21	0.040	Step
11-9 L	4	22	0.035	Fatigue
11-9 R	4	22	0.035	Step
11-11 R	5	19	0.034	Step
11-19 L	6	22	0.034	Step
11-26 L (1)	6	11	0.019	Step
11-30 L (1)	7	12	0.019	Step
12-6 B	7	24	0.038	Step
12-16	8	22	0.036	Step
12-17 BR	9	22	0.034	Tested twice, step 1st test to 1,000 psi
12-22 AL (1,2)	10	Note 2	0.015	100# step
12-22 AR (1,2)	10	Note 2	0.015	50# step

Notes: (1) Tapered transition wound onto ends of thin test specimens

(2) Ten layers - 2 axial layers between 4 inner and 4 outer circumferential layers

TABLE I

Internal Pressure Tests - 2-3/8" Diameter Microtape Tubes

I-B - Test Results

Tube Designation	Maximum Pressure, Water psi	Maximum Stress, 10^3 psi		Elastic Modulus, 10^6 psi	
		Axial	Circumferential	Axial	Circumferential
10-21 R	1500	None	42.7 no burst	None	9.5
10-21 R	1100	15.7	31.3	6.7	8.6
10-20	1100	18.8	37.6	8.6	9.8
10-26 L	1100	17.0	33.9	10.6	10.0
10-26 R	1550	24.5	49.0	-	-
10-27 R	250*				
11-5 R	900	15.5	31.0	-	10.3
11-5 L	600	10.3	20.6	13.3	10.4
11-8 L	1125	16.6	33.2	11.8	14.2
11-9 L	600	9.9	19.8	8.9	11.2
11-9 R	1100	18.2	36.4	13.5	11.4
11-11 R	800	13.7	27.4	10.2	16.0
11-19 L	1100	18.6	37.2	9.3	11.5
11-26 L	600	19.0	38.0	10.5	10.3
11-30 L	600	18.4	36.8	10.4	8.9
12-6 B	880**	13.6**	27.2**	14.2	10.1
12-16	1270	20.8	41.5	11.9	9.3
12-17 BR	1270	21.4	42.7	14.2	11.8
12-22 AL	450	17.0	34.0	-	-
12-22 AR	480	18.2	36.4	10.9	12.1
		Average step burst tests		Average omitting repeat on 10-21 R	
		17.8	35.6	11.3	11.2

* Poor structure

** not maximum stress, cap failure

TABLE II

Resin System Formulations

No.	
1	Epon 826 - 60 phr, DER 736 - 40 phr, HHPA - 81 phr, BDMA - 0.3%, Z-6040 - 2%
2	ERL 2256 - 100 phr, Sonite 41* - 27 phr, Z-6040 - 2%
3	Proprietary developmental epoxy-polyester resin
3a	Modification of "3" with increased catalyst and promoter
4	Proprietary developmental epoxy-polyester resin with styrene
5	DER 736 - 60 phr, Epon 826 - 40 phr, HHPA - 81 phr, BDMA - 0.3%, Z-6040 - 2%
6	Epon 826 - 80 phr, Epon 1001 - 20 phr, Tonox** - 25 phr, Z-6040 - 2%
7	Epon 826 - 80 phr, Epon 1001 - 20 phr, Tonox - 20 phr, MPDA - 5 phr, Z-6040 - 2%
8	Epon 826 - 80 phr, Epon 1001 - 20 phr, Tonox - 30 phr, MPDA - 7.5 phr, Z-6040 - 2%
9	Epon 826 - 80 phr, Epon 1001 - 20 phr, Tonox - 38 phr, MPDA - 9.5 phr, Z-6040 -2%
10	Epon 826 - 100 phr, MNA - 91 phr, BDMA - 0.3%, Z-6040 - 2%

* Sonite 41 - Smooth-On Manufacutring Co.

** Tonox - Naugatuck Chemical, Div. of U. S. Rubber Co.

TABLE III

DeBell & Richardson Microtape Results

Measured at Lewis Research Center

Specimen Designation		NOL Ring Tensile Test Stress psi x 10^3				Transverse* Shear psi	
		75°F	-320°	-423°	Glass Content by weight %	-320°F	75°F
A	1	104.3					
	2		131.5		89.6		
	4			149.2			
4-13-65B	1	115					
	2		144.7		90.5		
	3			140.8			
B	2	122.7				12,000	7,320
	4		155.7		91.5	11,200	7,790
						11,800	7,020
4-21-65A	1	117.0				8,230	7,900
	2	115.6				10,000	7,850
	3	116.8				12,600	8,600
	4		178.7			8,960	8,100
	5		164.0			13,400	8,230
	6		167.0			10,400	8,230
4-21-65B	1	120.3			88.7	7.090	
	2		171.7			9,370	
	3	112.2					
	4		173.0				
4-21-65C	1	102.7				13,600	
	2		131.5		90.3	10,000	
	3	103.0				7,260	
	4		129.3				

*Microtensile axial "dog biscuit" specimens.

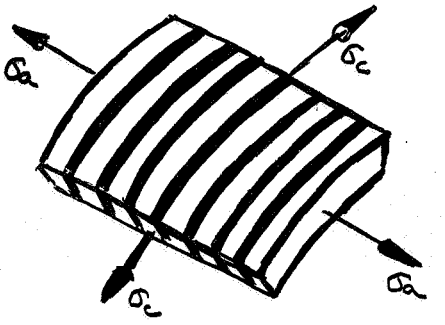
TABLE IV

Tensile Results of DeBell and Richardson Tubes
as Tested at Lewis Research Center

Tube No.	Temp. °F	Test	Burst P. psi	Tensile Load	Long. Tensile Strength, psi
11-19-65R	RT	Burst	1375		20,000
	RT	Tensile		5700	19,500
	RT	Burst	1380		20,100
11-8-65	RT	Burst	1113		16,100
		Tensile		4865	16,700
		Burst	1115		16,100
12-7-65B (#3)	RT	Tensile		5100	17,900
	LN_2	Tensile		7200	25,300
	RT	Burst	1235		
	RT	"(Rebuilt)	1450		22,700
	LN_2	Burst	1430		21,400
12-8-65A	RT	Tensile		4400	18,400
	LN_2	Tensile		4750	19,700
	RT	Burst	1230		
	LN_2	Burst	1360		
		New Tubes			
1-25-66A	RT	Burst	1145		17,900
	LN_2	Burst	1100		18,650
	RT	Tensile		5380	20,200
	RT	Tensile		5280	19,800
1-26-66B	RT	Burst	1380		22,500
	LN_2	Burst	1390		22,600
	RT	Tensile		6040	22,700
	LN_2	Tensile		5760	21,600
		6 Inch Diameter Tubes			
1-28-66A	RT	Burst	310		
1-21-66A	LN_2	Burst	240		

APPENDIX A

Analysis of Biaxial Stress Effects



In relating stresses and strains to arrive at an effective modulus, the Apparent Modulus, E' can be developed by dividing observed strains into the calculated stress based on the known loading:

$$E' = \sigma / e' \qquad (1)$$

In the general biaxial case,

$$\ell_1 = \ell_0 (1 + e') = \ell_0 \left(1 + \frac{\sigma_1}{E_1}\right)\left(1 - \frac{\mu\sigma_2}{E_2}\right) = \text{(approximately) } \ell_0 \left(1 + \frac{\sigma_1}{E_1} - \frac{\mu\sigma_2}{E_2}\right),$$

whence $e' = \sigma_1/E_1 - \mu\sigma_2/E_2$. (2)

For different materials cooperating in parallel to resist a load applied along the fibers of a composite, the amount of stretch, δ, is the same for all elements, and

$$\delta = \frac{P\ell}{AE} = \frac{P_t \ell}{A_t E'} = \frac{P_r \ell}{A_r E_r} = \frac{P_g \ell}{A_g E_g},$$

where sub-t = total structure, sub-r = resin elements, sub-g = glass elements, and the glass strands in the following discussion are assumed to lie in the circumferential direction. Assuming further that $E_g = 10^7$ psi, $E_r = 400,000$ psi, $A_r = .15A_t$, and $A_g = .85A_t$, the apparent circumferential modulus in unidirectional loading is

$$E_c = \frac{(P_r + P_g)\ell}{A_t \delta} = \frac{\ell}{A_t \delta}\left(\frac{A_r E_r \delta}{\ell} + \frac{A_g E_g \delta}{\ell}\right) = \frac{.15A_t E_r + .85 A_t E_g}{A_t} = 8,560,000 \text{ psi.}$$

For different materials cooperating in series to resist a unidirectional load applied perpendicular to the glass fibers, the stress is constant and the total deformation is the sum of the deformations of the glass and resin phases, assuming for the moment that the resin lies in continuous layers perpendicular to the applied load.

$$\delta = \frac{P_t \ell_r}{A E_r} + \frac{P_t \ell_g}{A E_g} = \frac{P_t \ell_t}{A E_a}; \quad E_a = \frac{P_t}{A} \times \frac{A}{P_t} \times \frac{\ell_t}{\ell_r/E_r + \ell_g/E_g} = 2,174,000 \text{ psi.}$$

When (as occurs in the pressure-testing of pipe with unrestrained ends) $\sigma_c = 2\sigma_a$, these moduli and stresses are into a synthesis of eqns. 1 and 2, above, the apparent axial and circumferential moduli of the assumed composite under the indicated biaxial stressing become (assuming $\mu = 0.3$)

$$E_a' = \frac{\sigma_a}{\sigma_a/E_a - 2\mu\sigma_a/E_c} = 2,564,000 \text{ psi, and } E_c' = \frac{\sigma_c}{\sigma_c/E_c - .5\mu\sigma_c/E_a} = 20,921,000 \text{ psi.}$$

When the resin layer is discontinuous, as it is in the microtape pipe, its deformation is markedly reduced, with corresponding increase in E_a' and decrease in E_c'. The effect is heightened by the distribution of substantial quantities of the resin in the plane parallel to the biaxial stress field, so that it is supported by the glass.